S-531

FINE GRAIN DENSE FERRITES

RAYTHEON

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Report No. 1

Contract No. DA36-039-AMC-00098(E)
DA Task No. 3A99-15-006-02

First Quarterly Progress Report

4 December 1962 - 3 March 1963

U.S. Army Electronics Research and Development Laboratory

Fort Monmouth, New Jersey

Research Division
Raytheon Company
Waltham 54, Massachusetts

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Object
To develop fine-grain dense ferrites for use at radio and microwave frequencies.

Prepared by

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1.0. PURPOSE

The purpose of the work performed under this contract is to develop and refine the techniques of preparation and evaluation of fine grained dense ferrites for use at both radio frequencies and microwave frequencies. Specifically, the radio-frequency applications are related to the temperature stabilized initial permeability that results from decreasing grain size while the microwave-frequency applications are related to the improved high-power characteristics that result from fine grain sizes.

2.0. ABSTRACT

The construction of a flame-spray apparatus was accomplished and compositions of $(Ni_{1-x}Zn_x)Fe_2O_4$ $0 < X < 0.75$ and $Ni(Fe_{2-x}Al_x)O_4$ $0 < X < 1.25$ prepared. X-ray analysis indicated single phase, stoichiometric compounds were formed in all cases. Hot pressed specimens of $NiFe_2O_4$ were prepared with grain sizes ranging from 5μ to below 0.2μ and ferrimagnetic resonance linewidths determined. The usual physical properties of ceramic specimens were determined for all samples.

Linewidth measurements were made on samples of $Ni_{0.973}Co_{.027}Fe_2O_4$ that were prepared by the Signal Corps.

3.0. PUBLICATIONS AND CONFERENCES

3.1 Publications

None.

3.2 Conferences

On January 22 W. W. Malinofsky and R. Babbitt visited the Raytheon Research Division to discuss the contract.

On February 14 R. Babbitt visited Raytheon and on February 28 J. J. Green, A. E. Paladino and J. S. Waugh visited Fort Monmouth to give a briefing on the progress of the contract.

3.3 Lectures

None.

3.4 Reports

None.

4.0. FACTUAL DATA

4.1 Flame-Spray Apparatus

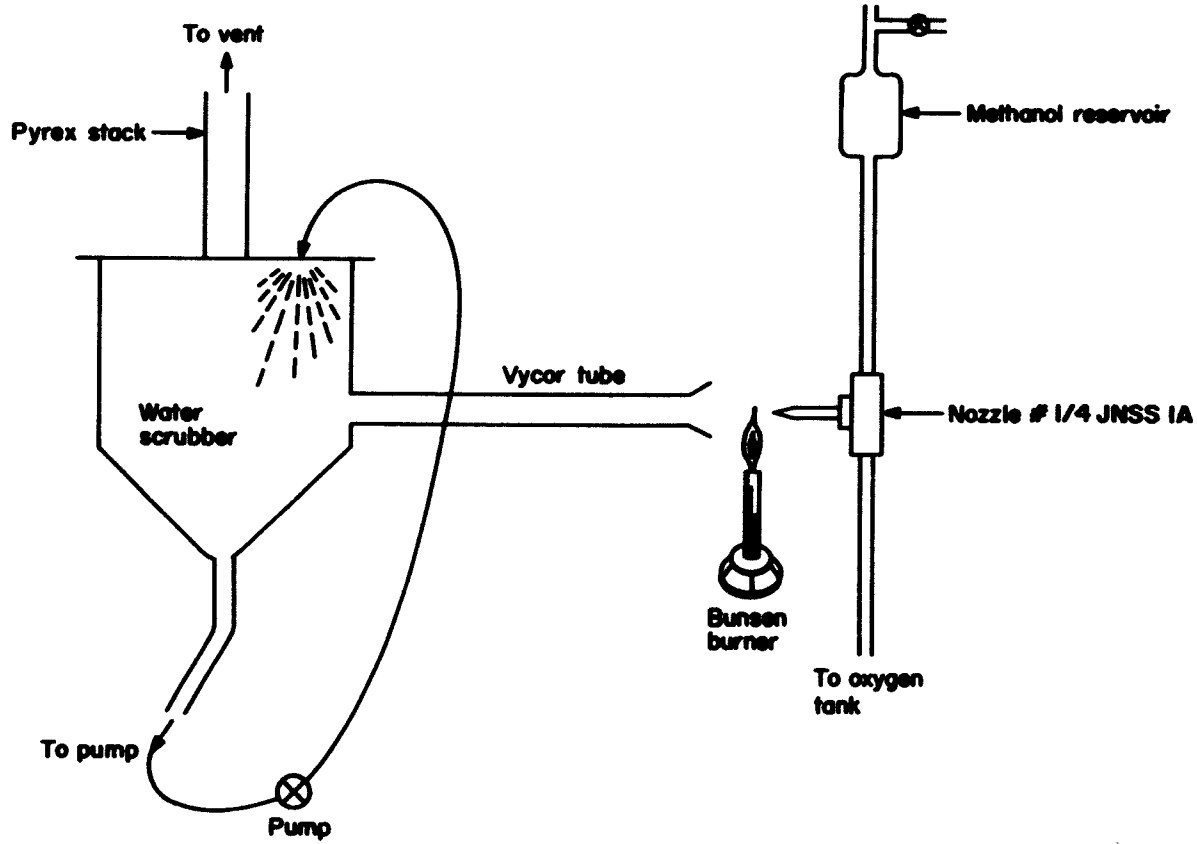
The flame-spray technique for the preparation of fine-particle sizes was used.[1,3] The sedimentation and chemical co-precipitation methods are available but their use is not anticipated.

The experimental set-up (fig. 1) consisted of an atomizer burner connected to tank oxygen and a methanol reservoir. The flame was maintained by a bunsen burner located at the mouth of a vycor tube which discharged into a large stainless steel tank. A water spray scrubber was placed at the top of the tank and effectively wet down the discharge from the vycor tube. Glass was used extensively throughout this system in order to (1) minimize the effects of contamination and (2) facilitate the cleaning of the system after each run (especially when the compositions were changed). A vertical pyrex stack guided the exhaust gases into a flue system. Yields greater than 60% of theoretical yield were obtained from this set-up and no contamination was evident in the resulting powders.

4.2 Preparation of Methanol Solutions

One molecular weight of the following nitrates was dissolved in one liter of methanol: $Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, $Zn(NO_3)_2 \cdot xH_2O$, and $Al(NO_3)_3 \cdot 9H_2O$. All easily dissolved with the exception of $Al(NO_3)_3 \cdot 9H_2O$ which showed some tendency to be cloudy. However, upon standing, the solution cleared and a sediment of Al_2O_3 collected on the bottom of the vessel.

Because of uncertainties in the amount of water of crystallization in the nitrates and because of generally good chemical procedure, all nitrate-methanol solutions were analyzed for oxide content in the following manner.



FLAME SPRAY APPARATUS
FIGURE 1

10 cc of the nominally one-molar solution were measured in a previously weighed platinum crucible. The solution was allowed to evaporate slowly and then ignited to 1000°C over a bunsen burner. Constant weights were considered achieved when no change occurred in the third decimal place. The reactions are described by the equations:

$$Ni(NO_3)_2 \cdot 6H_2O \xrightarrow{\Delta} NiO + 6H_2O + 2NO_2 + \tfrac{1}{2}O_2 \quad (1)$$

$$2Fe(NO_3)_3 \cdot 9H_2O \xrightarrow{\Delta} Fe_2O_3 + 18H_2O + 6NO_2 + \tfrac{3}{2}O_2 \quad (2)$$

$$Zn(NO_3)_2 \cdot xH_2O \xrightarrow{\Delta} ZnO + xH_2O + 2NO_2 + \tfrac{1}{2}O_2 \quad (3)$$

$$2Al(NO_3)_3 \cdot 9H_2O \rightarrow Al_2O_3 + 18H_2O + 6NO_2 + \tfrac{3}{2}O_2 \quad . \quad (4)$$

Following ignition, cooling, and weighing, the actual molarity of the solution was determined from simple calculation. Table I shows the variation in molarity that occurred for several batches of starting solution.

TABLE I

ANALYSIS OF NITRATE SOLUTIONS

Batch No.	Composition	Nominal Molarity	Actual Molarity
7384-28-123-1	$Ni(NO_3)_2 \cdot 6H_2O$	1	0.965
7384-28-123-2	$Fe(NO_3)_3 \cdot 9H_2O$	1	0.976
7384-34-212-1	$Zn(NO_3)_2 \cdot xH_2O$	1	1.052
7384-34-212-2	$Al(NO_3)_3 \cdot 9H_2O$	1	0.588
7384-28-123-3	$Ni(NO_3)_2 \cdot 6H_2O$	1	0.976
7384-28-123-4	$Fe(NO_3)_3 \cdot 9H_2O$	1	0.963
7384-34-212-3	$Zn(NO_3)_2 \cdot xH_2O$	1	1.066
7384-34-212-4	$Al(NO_3)_3 \cdot 9H_2O$	1	0.665
7384-35-213-1	$Ni(NO_3)_2 \cdot 6H_2O$	1	0.987
7384-35-213-2	$Fe(NO_3)_3 \cdot 9H_2O$	1	0.965
7384-34-212-5	$Zn(NO_3)_2 \cdot xH_2O$	1	1.052
7384-34-212-6	$Al(NO_3)_3 \cdot 9H_2O$	1	0.673
7384-39-225-1	$Ni(NO_3)_2 \cdot 6H_2O$	1	0.843
7384-39-225-4	$Fe(NO_3)_3 \cdot 9H_2O$	1	0.912
7384-39-225-3	$Al(NO_3)_3 \cdot 9H_2)$	1	0.616

4.3 Preparation of Ferrites

The following compositions, all of which were single phase and stoichiometric as determined by standard X-ray analysis, were prepared

during this quarter:

$(Ni_{1-x}Zn_x)Fe_2O_4$ $0 \leq X \leq 0.75$

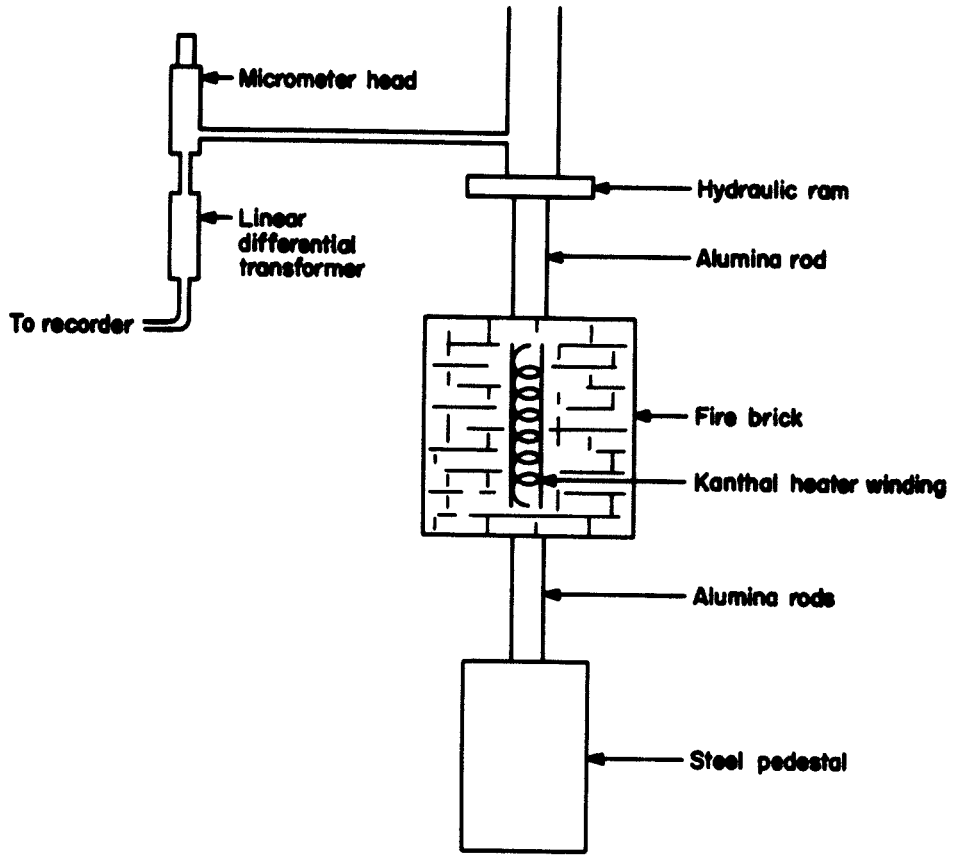
increment of $x = 0.25$

$Ni(Fe_{2-x}Al_x)O_4$ $0 \leq X \leq 1.25$.

The procedure consisted of measuring, by volume, the constituent solutions in the ratios consistent with stoichiometry previously determined by analysis of the starting solutions. The liquids were mixed in a flask and poured into the methanol reservoir connected to the atomizer burner. The oxygen pressure was maintained at 20 psi (by means of a standard tank regulator) and the needle valve opening recorded. The hydrostatic head was also noted and kept constant as was the distance from the nozzle to the burner flame. All of these variables affect the particle size of the resultant powder. (R. Babbitt pointed out the possible pitfalls in this procedure.) The powder was collected in the water spray system as a slurry and the entire contents of the run was collected and dried for use. This included the dry powder in the vycor and pyrex tubes as well as the slurry collected in the stainless steel tank. No evidence of nitrates was found in the dried powder.

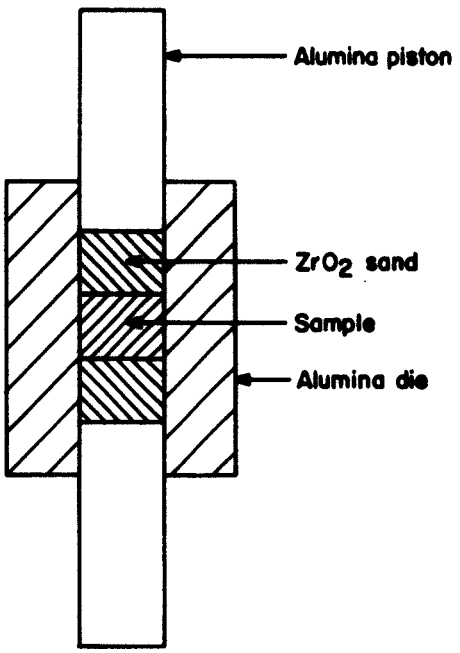
4.4 Hot Pressing

The hot pressing of the ferrites (fig. 2) was done in hot-pressed alumina dies obtained from the Carborundum Company of Niagara Falls, New York. Many variations were tried, but the best technique consisted of prepressing the ferrite powder in the die, removing the sample, sanding off the edges so that it fit loosely in the die and finally covering both ends of the specimen with ZrO_2 sand (-325 mesh). The ZrO_2 sand prevented sticking to the pistons and the fine particle size was essential in order to transmit the load from the pistons to the sample. Larger sized grains of ZrO_2 cannot slide over one another easily and consequently the load would not be fully applied to the sample. (fig. 3).



HOT PRESS

FIGURE 2



HOT PRESS DIE

FIGURE 3

The force required for hot pressing was applied from a hydraulic ram which was continuous in operation. The ram was controlled by hydraulic pressure which was independent of the movement of the piston; consequently, the pressure within the die was constant throughout the densification process. The course of densification was continuously recorded by means of a linear differential transformer which was electrically connected to a strip recorder. By this procedure, the entire densification process was monitored so that complete densification could immediately be observed. A typical hot-press strip chart is shown in fig. 4. In all cases, final pressures were 12, 600 psi; during heat up, the pressure was either 3600 or 7200 psi.

The hot-press die was heated resistively by means of a kanthal wound ceramic tube insulated by refractory brick. This set-up achieved a heat-up time of 10 minutes.

4. 5 Physical Property Measurements

4. 5. 1 Density

All density determinations were made by the mercury displacement method which measures the bulk volume of the sample.

Table II lists the densities, grain sizes, and composition of all the hot-press specimens. A rather significant result shows that considerable grain growth occurred in nickel ferrites at temperatures as low as 1050°C when the initial particle size is sub-micron.

HOT PRESS DENSIFICATION CURVE

FIGURE 4

TABLE II

Grain Size, Density and Firing Conditions
For $NiFe_2O_4$ and $(Ni_{0.5}Zn_{0.5})Fe_2O_4$

Sample	Final Grain Size	Bulk Density	Apparent Density	Firing Temp.	Firing Time at Temp.
$NiFe_2O_4$					
7384-41-1	3.5μ	88.2%	96.2%	1125°C	20 min.
7369-25-1	~5μ	98.0%	99.0%	1125°C	30 min.
7369-25-2	3μ	90.0%	97.0%	1140°C	10 min.
7369-30-1	0.5μ	97.3%	99.0%	1050°C	10 min.
7369-25-3	1.0μ	87.0%	94.0%	1050°C	15 min.
7384-43-1	0.2μ	99.0%	99.0%	1025°C	3 min.
7384-43-3	0.3μ	99.0%	99.0%	1025°C	5 min.
7384-43-2	0.22μ	85.0%	91.0%	1025°C	3 min.
$(Ni_{0.5}Zn_{0.5})Fe_2O_4$					
7369-26-1	1μ	83.0%	90.5%	1125°C	30 min.

4.5.2 Grain size

The grain size of the powder received from the flame spray was determined by (1) X-ray line broadening techniques and (2) electron microscopy.

(1) The X-ray line broadening technique is essentially one outlined in Klug and Alexander.* A known standard was used, designated NFK-O, which was well annealed, stoichiometric $NiFe_2O_4$. The instrumental as well as the mosaic broadening, β, was determined for the standard. The formula $L = \frac{K\lambda}{\beta \cos\theta}$ was used where

L = crystallite size (A)
λ = radiation wavelength
β = broadening due to particle size
K = shape factor .

The unknown specimens were then compared to the standard and the difference in the peaks used for assessing the broadening due to particle size. Strain broadening was neglected. This method is admittedly crude, but nevertheless adequate for the approximate determination of these grain sizes.

(2) An RCA EML-1 was used for the electron microscopy. For determination of the particle size of the flame-spray powder, a slurry technique was used where the powder was suspended in a solution of methyl alcohol (anhydrous). The agglomerates were broken up by use of an ultrasonic agitator. The suspension was allowed to evaporate onto a carbon film that had previously been deposited onto a supporting grid. Figure 5 illustrates the technique for a specimen of $NiFe_2O_4$. The particle size was estimated to be between 0.05 - 0.09, in fair agreement with X-ray determinations (see Table III).

It was generally found that the greatest difficulty was to minimize the grain growth of $NiFe_2O_4$ rather than achieve the maximum density. One

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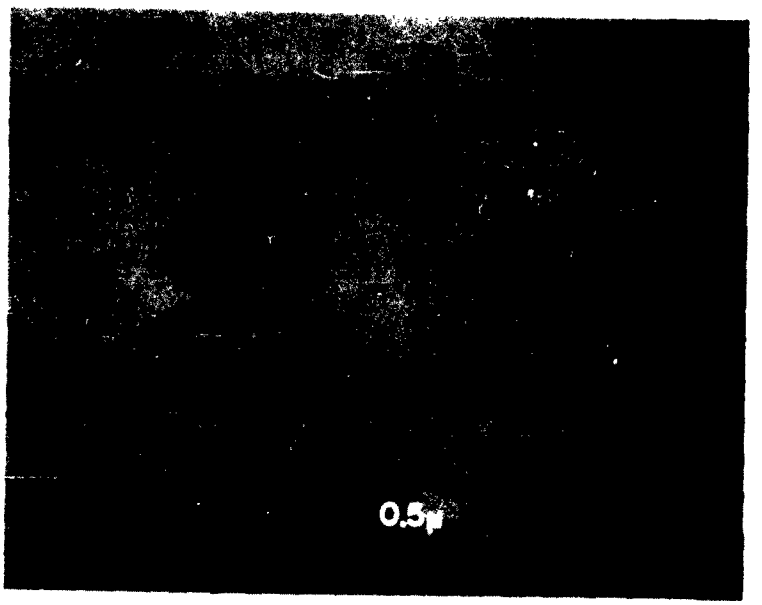
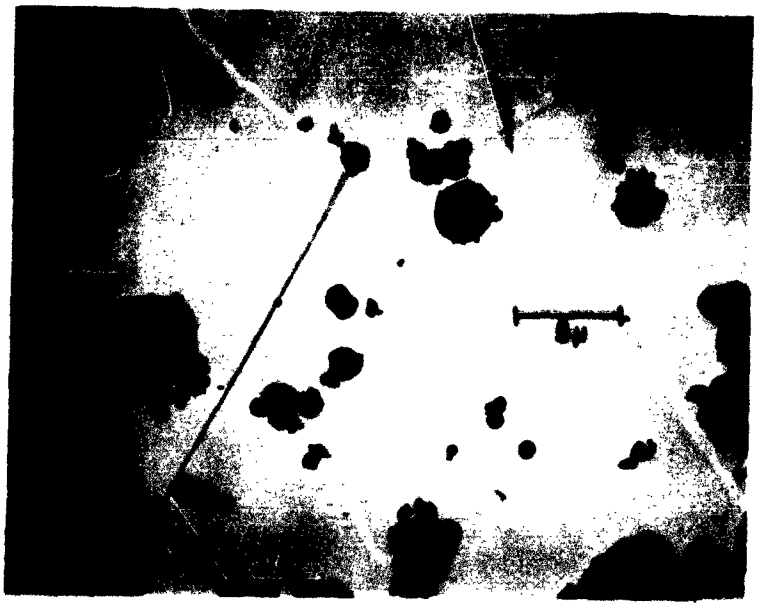
*H. P. Klug and L. E. Alexander, X-Ray Diffraction Procedures, John Wiley and Sons, Inc., New York (1954) pp. 507-511.



$NiFe_2O_4$ 27,000X



$NiFe_2O_4$ 2000 X

ELECTRON MICROGRAPHS OF FLAME SPRAYER $NiFe_2O_4$

FIGURE 5

TABLE III

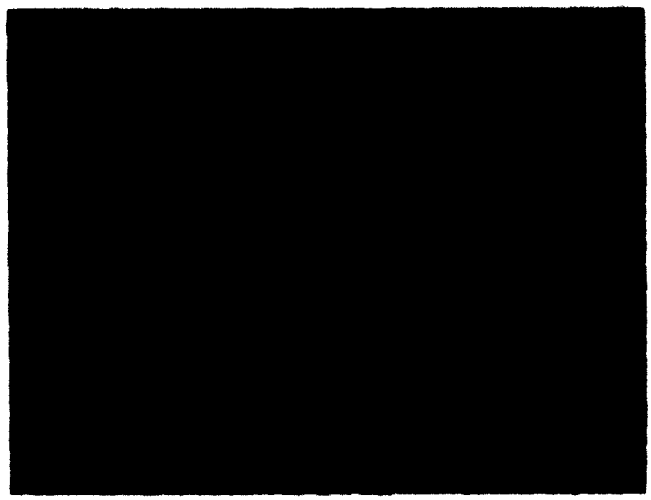
Comparison of X-Ray and Electron Micrograph
Particle Size Determinations

Sample	Particle Size (X-ray) (μ)	Particle Size (Electron Microscope) (μ)
$NiFe_2O_4$		
7369-25	0.06	0.05
7384-43	0.07	0.065
7384-33	0.085	0.08
$(Ni_{0.75}Zn_{0.25})Fe_2O_4$		
7384-35	0.09	0.085

of the first samples to be hot-pressed is shown in fig. 6. The sample was polished with Cr_2O_3 on a silk wheel and etched in a mixture of oxalic and sulfuric acid for six minutes. The grain size was relatively large (5μ), and the density (bulk) was greater than 98% of the theoretical density. It is also interesting to note where the remaining porosity was located. In almost all cases, the pores were situated at the grain boundaries rather than within the grains.

4.5.3 Stoichiometry

The stoichiometry of $NiFe_2O_4$ was carefully substantiated by means of comparison with a known stoichiometric sample of $NiFe_2O_4$ designated NFK-0. It was deemed necessary to evaluate the $NiFe_2O_4$ powder being made



$NiFe_2O_4$ 800X

POLISHED SECTION OF HOT PRESSED $NiFe_2O_4$

FIGURE 6

in the flame-spray apparatus in order to determine this stoichiometry. This was done by a three-way comparison of the relative intensity ratios between the unknown, the NFK-0, and a theoretical calculation for spinel structure.

The quality of the standard was substantiated by a comparison of the relative intensity ratios with a series of intensity ratios calculated from the formulae:

$$F_{hk\ell} = \sum_{n=1}^{N} f_n e^{2\pi i(hx_n + ky_n + \ell z_n)}$$

$$I = |F|^2 \times \text{Lorentz Polarization Factor} \times \text{Multiplicity} \quad .$$

This comparison is shown in Table IV.

TABLE IV

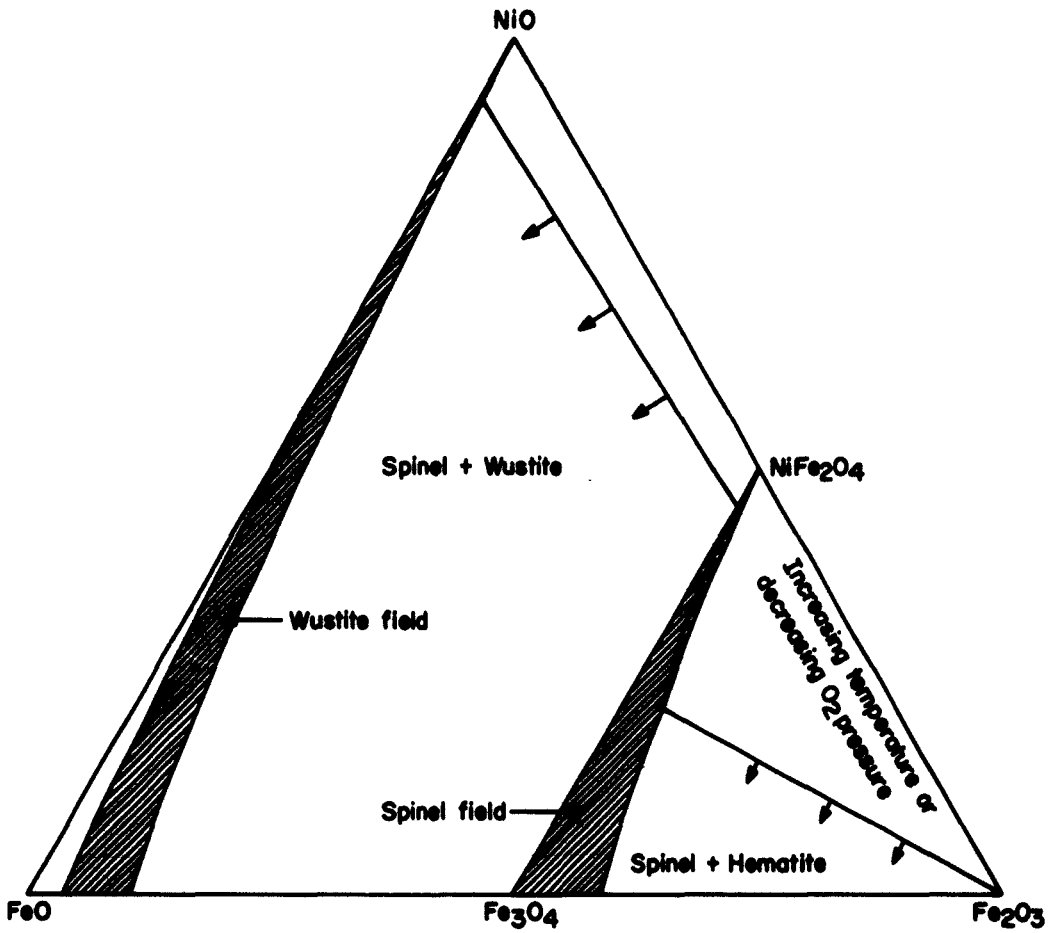
Calculated and Measured X-Ray Intensity Data

$hk\ell$	$\lvert F_{hk\ell} \rvert^2$	m	LP	I	Calc. I/I_1	Measured I	Measured I/I_1
111	7648	8	55.33	70527	14	147	15
220	28009	12	19.02	133183	27	288	29
311	75024	24	13.30	498910	100	979	100
222	23446	8	11.93	46618	9	65	7
400	102810	6	8.458	108696	22	285	29
331	845	24	6.783	2866	< 1	20	2
422	17742	24	5.128	45490	9	82	8
511	66744	24	4.445	177412	36	316	32
333	39244	8	4.445	177412	36	316	32
440	245223	12	3.660	224379	45	440	45
531	2810	48	3.327	9349	2	15	2

The diffraction pattern of nickel ferrite samples prepared early in the program revealed departures from the expected peak intensity ratios. Past experience indicated that this might be caused by the presence of a nickel oxide-rich wüstite phase formed during a partial reduction of the $NiFe_2O_4$ (fig. 7).[4] For starting compositions lying between $NiFe_2O_4$ and NiO, a partial reduction would correspond to the formation of a spinel solid solution of $NiFe_2O_4$-Fe_3O_4 plus a solid solution of (NiFe)O high in nickel content. The situation is best described by a typical tie line in fig. 7 illustrating the changes in composition of both the spinel and wüstite phases with progressively more reduction (indicated by the arrows on the tie). The lattice constant for solid solutions between $NiFe_2O_4$ and Fe_3O_4 varies linearly so that it was used as a check on the extent of reduction, if any.

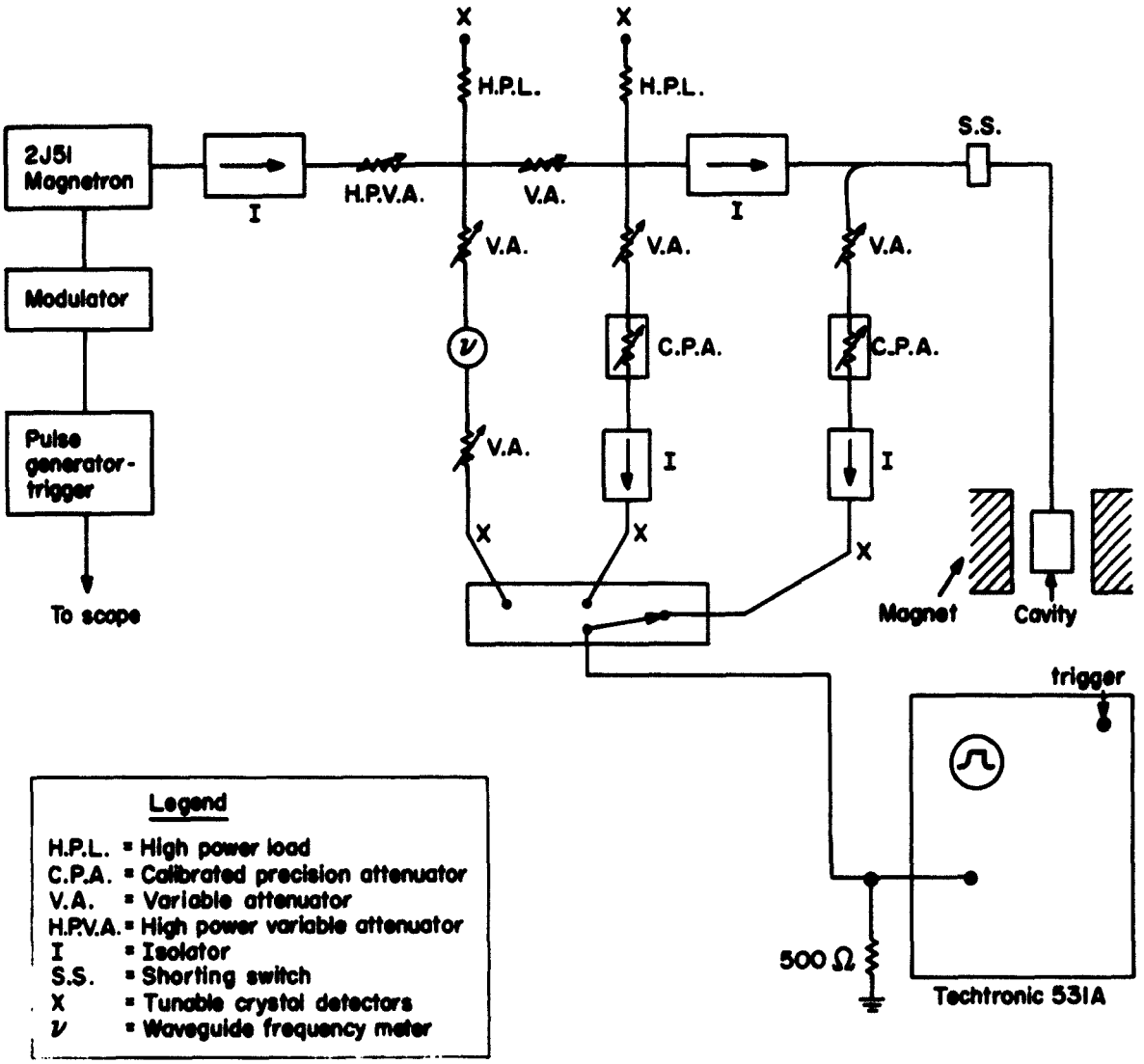
4. 5. 4 High-power subsidiary absorption and linewidth measurements

Figure 8 is a schematic diagram of the apparatus used in making high-power measurements at X-band. By a 90° rotation of either magnet or cavity the parallel pumping or the subsidiary absorption measurement can be made. A Raytheon 2J51 tunable magnetron was used as a signal source. This magnetron delivers a peak power of 40 kw which was sufficient to produce rf cavity fields in excess of 100 oe. Pulse lengths of 4 μ sec and a repetition rate of approximately 6 cps were used. This low-duty cycle insured that no sample heating occurred. The power incident upon the cavity was controlled through several variable attenuators in the main line. The incident power and reflected power were monitored through directional couplers preceding the cavity. A power calibration was performed by replacing the cavity by a thermistor, the magnetron by a square-wave modulated klystron, and then noting the power-meter reading for a given deflection on the oscilloscope from the crystal which samples the incident power. All other power levels were referred to this level through the use of a calibrated precision attenuator (rotary-vane type) which precedes the crystal. A knowledge of the incident power, the loaded Q of the cavity, the cavity reflection coefficient, and the cavity mode then gave the value of the rf magnetic field at the sample.



PHASE RELATIONS IN THE SYSTEM $FeO-Fe_2O_3-NiO$

FIGURE 7



EXPERIMENTAL APPARATUS FOR HIGH POWER MEASUREMENTS AT X BAND

FIGURE 8

The subsidiary absorption measurement was made by measuring the cavity reflection coefficient, and therefore the cavity Q as a function of power level.

The sample (a sphere approximately 50 mil diameter) was mounted several sphere diameters from the end wall if a TE_{102} reflection cavity.

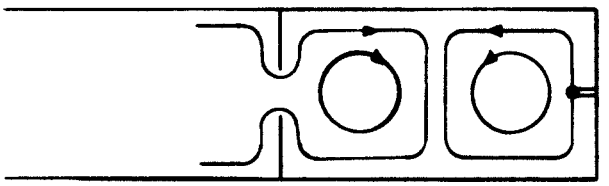


Figure 9
Cavity for high-power subsidiary absorption measurements.

A similar cavity was used for linewidth measurements except that it was of a transmission type and the sample was mounted in the center of the cavity. In linewidth measurements 30-mil spheres were usually sufficient since the absorption was much larger in the vicinity of ferromagnetic resonance than away from it as in the case of subsidiary absorption.

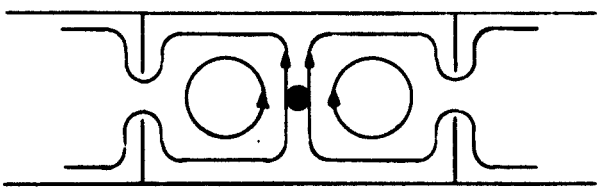
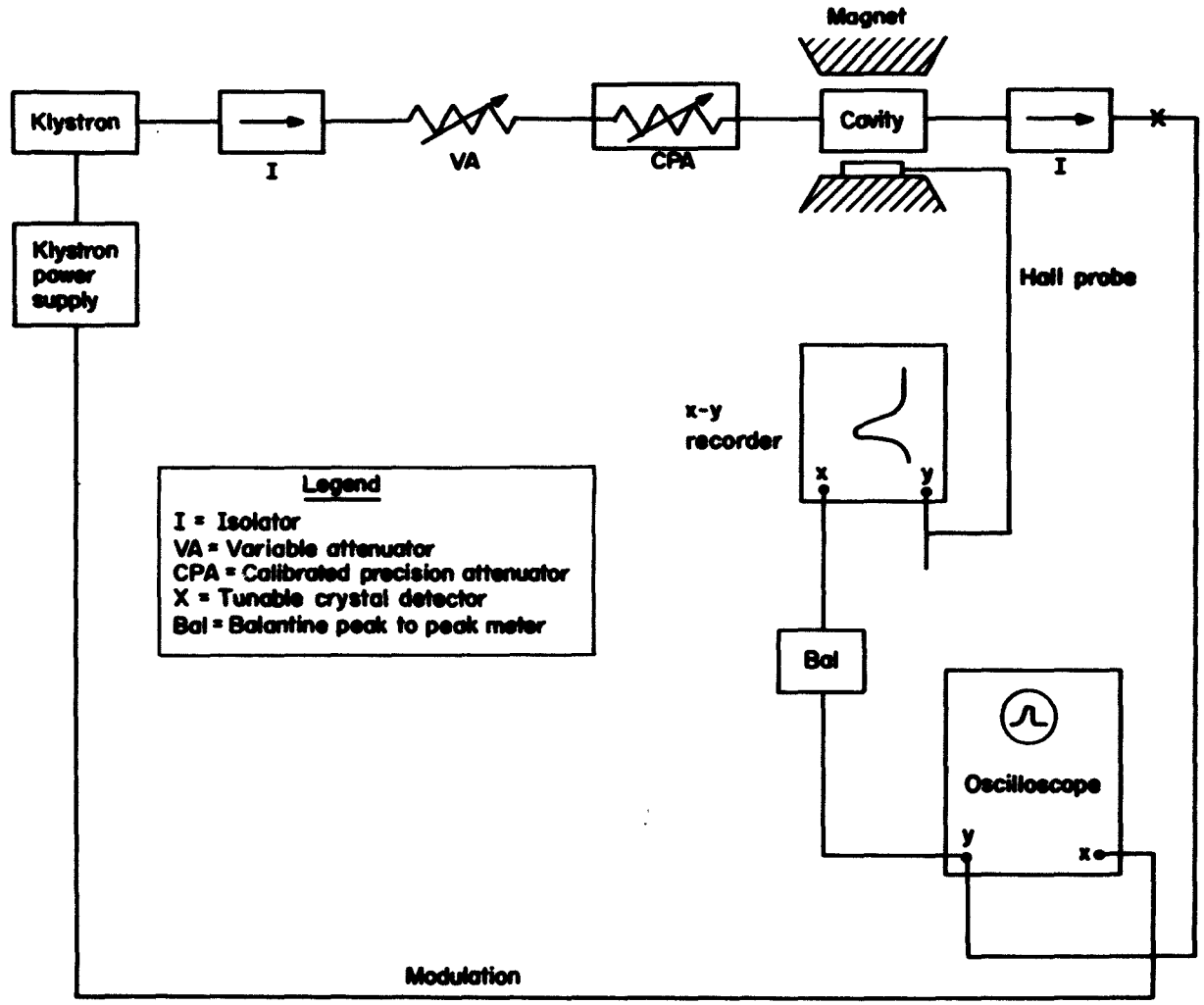


Figure 10
Cavity for linewidth measurement.

Figure 11 is a schematic diagram of the linewidth equipment. A saw tooth modulated klystron was used to sweep through the cavity mode. A Ballantine voltmeter was used to record the peak value of the cavity transmission on the X-axis of an X-Y recorder.



LINE WIDTH MEASUREMENTS AT X BAND

FIGURE II

The Y-axis of the recorder corresponds to the dc field and was driven by a signal from a Hall probe in the magnet gap. By sweeping the dc field the resonance line was automatically traced on the recorder. The linewidth was determined through the use of a calibrated precision attenuator which preceded the cavity and which permitted an accurate determination of the change in cavity transmission.

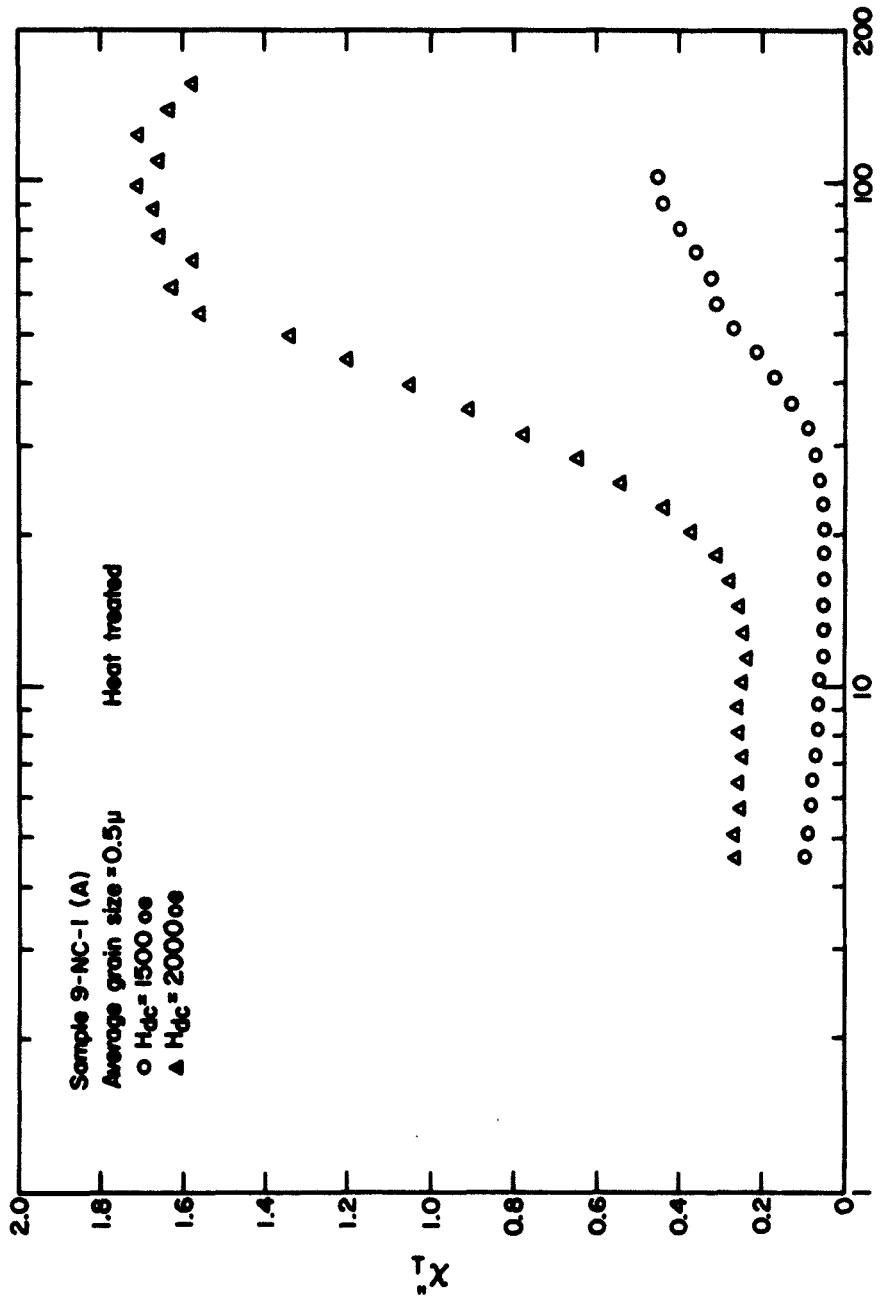
Linewidth and subsidiary absorption measurements were made at X-band (9.2 kMc) on some heat treated fine-grain nickel-cobalt ferrites (2.7% Co). These samples were prepared by W. W. Malinofsky and R. W. Babbitt at the U. S. Army Electronics Research and Development Laboratory. The data are compiled below, and are shown in figs. 12-15.

TABLE V

Linewidth and Grain Size Data for Signal Corps $(Ni_{0.973}Co_{0.027})Fe_2O_4$ Samples

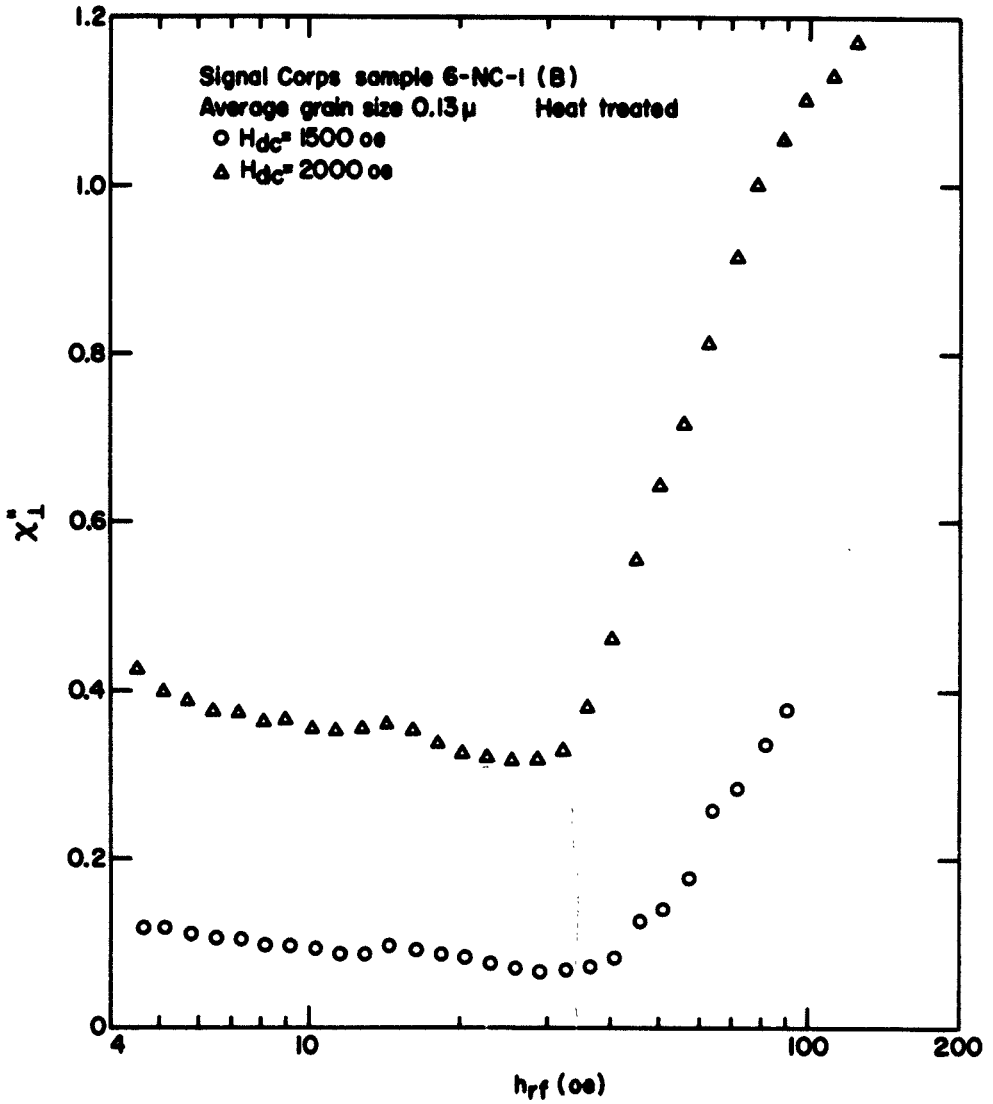
Sample	Grain Size (μ)	$\Delta H_{(1)}$ (oe)	$\Delta H_{(2)}$ (oe)	Subsidiary Absorption $H_{dc(1)}$ (oe)	$h_{rf(1)}$ (oe)	$H_{dc(2)}$ (oe)	$h_{rf(2)}$ (oe)
4-NC-1 (A)	0.18	565	485	1500	none	1500	38
				2000	none	2000	31
6-NC-1 (A)	0.25	646	470	1500	60	1500	40
				2000	50	2000	35
6-NC-1 (B)	0.13	671	415	1500	55	1500	40
				2000	45	2000	31
9-NC-1 (A)	0.50	1042	335	1500	42	1500	30
				2000	35	2000	17

In order that a comparison might be drawn, date for "before heat treatment" has been included and is designated by subscript (1). The h_{rf} values listed are for the thresholds for absorption below resonance. The measurements were made by setting a particular H_{dc} value while varying the h_{rf} level in the cavity.



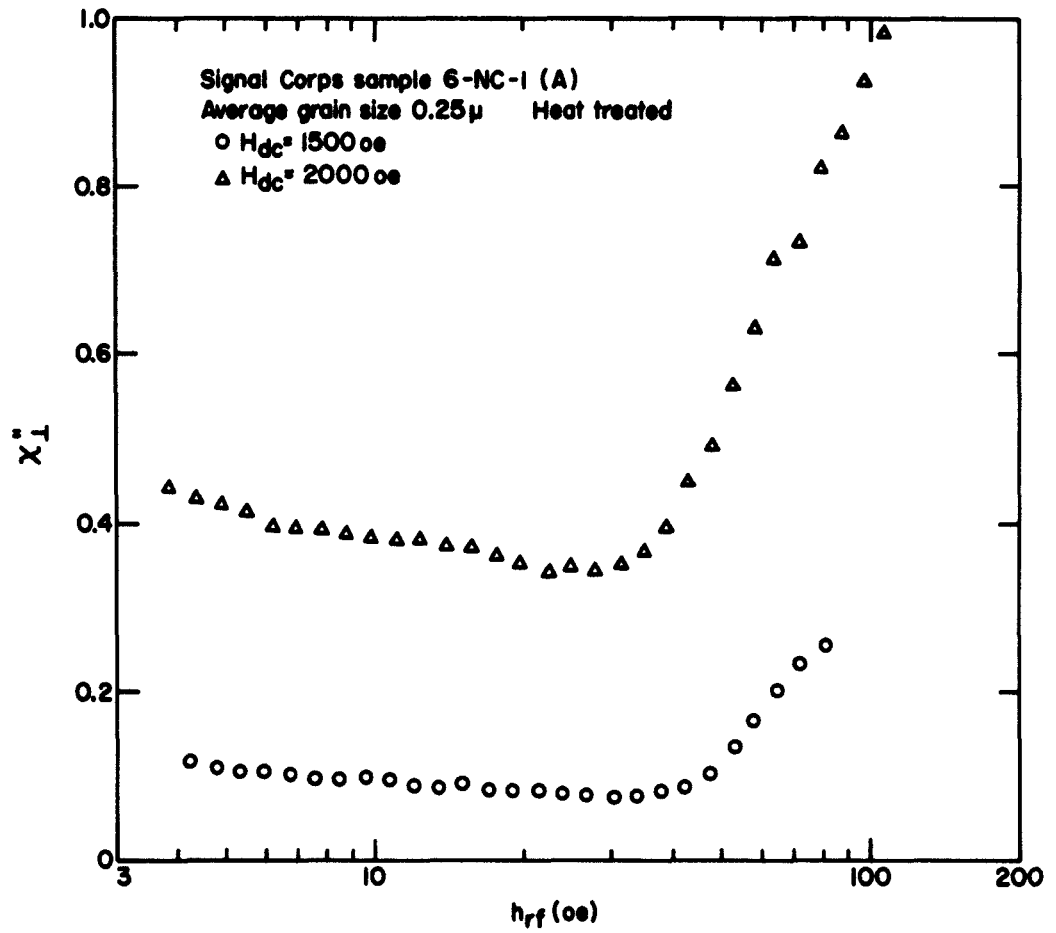
SUBSIDIARY ABSORPTION SUSCEPTIBILITY VS R.F. MAGNETIC FIELD

FIGURE 12



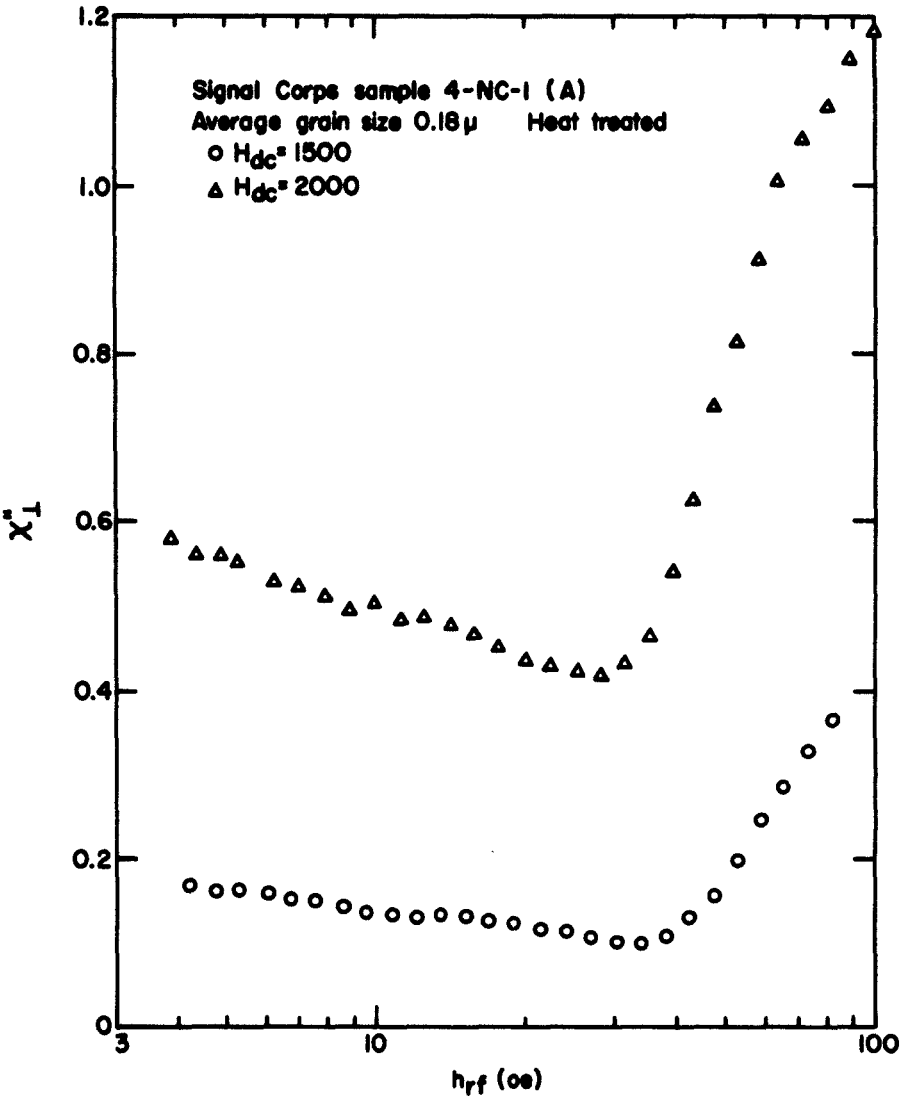
SUBSIDIARY ABSORPTION SUSCEPTIBILITY VS R.F. MAGNETIC FIELD

FIGURE 13



SUBSIDIARY ABSORPTION SUSCEPTIBILITY VS. R.F. MAGNETIC FIELD

FIGURE 14



SUBSIDIARY ABSORPTION SUSCEPTIBILITY VS. R.F. MAGNETIC FIELD

FIGURE 15

Below is a compilation of linewidths of the latest fine-grain ferrites:

TABLE VI

Linewidth and H_{res} Data for Hot Pressed $NiFe_2O_4$

Sample	Identification	H_{res} (oe)	ΔH (oe)
$NiFe_2O_4$	7369-21-3	2941	1698
$NiFe_2O_4$	7369-21-2	3060	2520
$NiFe_2O_4$	7369-21-1	2928	2742
$NiFe_2O_4$	7384-35-1	3004	2499
$NiFe_2O_4$	7369-25-1	3361	570
$NiFe_2O_4$	7369-25-2	3469	567
$NiFe_2O_4$	7369-25-4	3459	1383
$NiFe_2O_4$	7369-25-3	3417	1286

5.0 CONCLUSIONS

5.1 Flame-Spray Prepared Ferrites

The flame-spray technique for producing single-phase stoichiometric ferrites yielded materials in the desired particle size range (sub-micron < 0.05). Although many variables affected the final particle size, it was not difficult to isolate most of them so that consistent results were obtained. The technique was also useful for the production of mixed ferrites where one of the constituents was more volatile than others (Zn).

5.2 Hot Pressing of $NiFe_2O_4$

$NiFe_2O_4$ can successfully be hot pressed to densities approaching its theoretical (> 98%) density. The grain size within the hot pressed specimen however, exceed the previously established results. For example, at pressing temperatures as low as 1025°C for times as short as three minutes, the grain size of stoichiometric $NiFe_2O_4$ (initial particle size of 0.02μ) is consistently above 0.2 μ.

6.0 PLANS FOR NEXT QUARTER

Further investigation of the hot pressing of $NiFe_2O_4$, $(Ni_{1-x}Zn_x)Fe_2O_4$, and $Ni(Fe_{2-x}Al_x)O_4$ will be pursued with the hope of achieving dense fine-grained specimens of all of these compositions. The subsidiary absorption linewidth, high-power properties, temperature dependence of initial permeability, dielectric loss, saturation magnetization, density, and grain size will be measured on the appropriate specimens.

Compositions with the garnet structure will be prepared.

7.0 IDENTIFICATION OF PERSONNEL

Below is a list of the personnel involved in this investigation and the amount of time each spent on the project:

E. Schlömann - Principal Research Scientist	Consultant
J. S. Waugh - Senior Research Scientist	140 hours
W. Bekebrede - Senior Research Scientist	86 hours
A. E. Paladino - Senior Research Scientist	16 hours
J. J. Green - Principal Research Scientist	21 hours
B. J. Healy - Associate Research Scientist	72 hours
T. Kohane - Senior Research Scientist	16 hours
O. J. Guentert - Principal Research Scientist	32 hours
Total	383 hours

Following is a list of the biographies of the above-mentioned personnel involved in this investigation.

Ernst Schlömann - Principal Research Scientist

Dr. Schlömann received his M.S. in 1953 and his Ph.D. in 1954, both in theoretical physics, from the University of Goettigen, Germany. He served as a Research Assistant for one year at this institution. From 1954 to 1955 he did post-doctoral work in theoretical solid state physics at MIT.

In 1955 Dr. Schlömann became a full-time member of the Research Division of Raytheon Company. His field is the theoretical study of new ferromagnetic materials and their application to microwave devices. He has made numerous important contributions to the theory of resonance linewidth in polycrystalline materials and to the theory of ferromagnetic resonance at high signal levels. During the academic year 1961 - 1962 Dr. Schlömann served as a visiting associate professor at the W.W. Hansen Microwave Laboratory, Stanford University. He returned to Raytheon in June 1962 to resume his research on ferromagnetic materials.

Dr. Schlömann is a member of the American Physical Society.

John S. Waugh - Senior Research Scientist

Dr. Waugh received a B. S. in 1953 and an M. S. in 1954 both in ceramics from Alfred University. During 1954 - 1955, he was on the chemical and metallurgical program at General Electric Company. In January, 1960, he was awarded a Ph. D. in ceramics from M. I. T.

During 1960, Dr. Waugh was a section head in the Physics Group of ITEK Corporation. In January, 1961, he joined the Raytheon Research Division as a research staff member. His main activity has been research on new ferroelectric materials. He has been experimenting with the growth of single crystals of barium-strontium titanate.

Dr. Waugh is a member of the American Ceramic Society, the American Physical Society and Sigma Xi.

Wilfred R. Bekebrede - Senior Research Scientist

Mr. Bekebrede received his A.B. degree in chemistry from Washington University in 1947. Ohio State University awarded him an M.S. in inorganic chemistry in 1952. He has completed advanced courses in chemistry, mathematics, and electronics at the Northeastern University Evening Graduate School of Engineering and at Boston University. He also attended a course in X-ray diffraction and X-ray spectroscopy techniques at the North American Phillips Company.

From 1947-1950 Mr. Bekebrede was an analytical chemist for Monsanto Chemical Company, Monsanto, Illinois. Subsequent to receiving his master's degree he did research and development work on electrolytes for the Fansteel Metallurgical Corporation, North Chicago, Illinois. In 1954, Mr. Bekebrede joined the Research Division of Raytheon Company, where he is now utilizing X-ray diffraction techniques in the study of electronic materials.

Mr. Bekebrede is a member of the American Crystallographic Association.

Albert E. Paladino, Jr. - Senior Research Scientist

Dr. Paladino received his B.S. in ceramic engineering in 1954, and his M.S. in the same field in 1956, from the New York State College of Ceramics, at Alfred University. In 1956 and 1957 he did additional work at the Northeastern University Evening Graduate School of Engineering.

Dr. Paladino joined the Research Division in 1955 where, until 1959, he investigated the high temperature chemistry of ferrites. Portions of the phase diagrams for the systems Fe-Ni-O, Fe-Co-O, and MgO-FeO-Fe_2O_3 in the area of spinel have been determined over a range of temperatures and oxygen pressures.

In 1959, Dr. Paladino entered the Department of Metallurgy, Division of Ceramics, at MIT where he started graduate study for his doctor's degree. His thesis research was concerned with aluminum ion diffusion in aluminum oxide. In January 1962, he received his Doctor of Science degree from MIT and returned to the Research Division where he assumed the responsibility of directing the research activities of the ceramic laboratory.

Dr. Paladino is a member of the American Ceramic Society, American Physical Society, Keramos, and is a registered professional engineer in the Commonwealth of Massachusetts.

Jerome J. Green - Principal Research Scientist

Dr. Green received his B.S. in physics from Northwestern University in 1954. His A.M. in solid state physics was received in 1955 and his Ph.D. in 1959, both from Harvard University. His thesis dealt with ferromagnetic resonance effects in ferrites and garnets at high signal levels.

From 1956 to 1959, Dr. Green worked part time at the Raytheon Research Division. Upon receipt of his doctorate in June 1959, he joined the Company on a full-time basis. At Raytheon Dr. Green has been engaged in research concerning all types of high power effects in ferromagnetic materials at microwave frequencies.

Barbara Healy - Associate Research Scientist

Miss Healy received a B.S. in mathematics and physics from Simmons College in 1956. In August of that year she joined the staff of the Raytheon Research Division.

During her six years experience, Miss Healy's work has included the fields of paramagnetic resonance and plasma physics. Recently she has been working on ferrite measurements, including the study of fine-grain materials.

Theodore Kohane - Senior Research Scientist

Dr. Kohane received the degree of B. S. in physics from the College of the City of New York in 1944. From 1944 to 1946 he was employed as a physicist with the National Advisory Committee on Aeronautics at Langley Field, Virginia. He then undertook graduate study at New York University and later Rutgers University, holding various teaching and research assistantships and fellowships. In 1953 he received the degree of Ph. D. in physics from Rutgers University; his thesis was concerned with nuclear magnetic resonance studies of hydrogen in palladium.

Since 1953 Dr. Kohane has been a member of the Research Division staff. After some work on silicon and semiconducting compounds, he became engaged in studies of the magnetic and electrical properties of ferrimagnetic oxides, and is currently working in this area.

Dr. Kohane is a member of the American Physical Society and of the Microwave Ferrite Group of the A. S. T. M.

Otto J. Guentert - Principal Research Scientist

Dr. Guentert received his diploma in physics from the University of Freiburg, Germany, in 1951, and his Ph. D. in physics (x-ray diffraction) from MIT in 1956.

From 1953 to 1956 he was a research assistant in the Physics Department of MIT where he worked under Prof. B. E. Warren on x-ray diffraction problems of cold work and layer faults in metals.

Dr. Guentert joined the Solid State Physics Group of the Raytheon Research Division on July 1, 1956. He is a principal research staff member in charge of the diffraction laboratory which supports the activities of the Research Division by x-ray and neutron investigations. He is presently engaged in diffraction studies of several materials. The research projects include (a) the determination of the cation distribution in mixed spinel ferrites; (b) the study of the structural features of PYROGRAPHITE; and (c) the study of the layer ordering in ZnS-type crystals, notably SiC. Dr. Guentert would carry out the x-ray diffraction part of the bicrystals program

Dr. Guentert is a member of the American Physical Society and of the American Crystallographic Association.

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ULTRA FINE GRAIN DENSE FERRITES by
J. S. Waugh, A. Paladino and others.
Quarterly Report No. 1, 1 Dec 1962 - 28 Feb 1963
41 p. incl. illus. (S-531)
(Contract DA 36-039-AMC-00098 (E))
Unclassified Report

The construction of a flame-spray apparatus was accomplished and compositions of $(Ni_{1-x}Zn_x)Fe_2O_4$ $0 \leq X \leq 0.75$ and $Ni(Fe_{2-x}Al_x)O_4$ $0 \leq X \leq 1.25$ prepared. X-ray analysis indicated single phase, stoichiometric compounds were formed in all cases. Hot pressed specimens of $NiFe_2O_4$ were prepared with grain sizes ranging from 5μ to below 0.2μ and ferrimagnetic resonance linewidths determined. The usual physical properties of ceramic specimens were determined for all samples.
Linewidth measurements were made on samples of $Ni_{0.973}Co_{0.027}Fe_2O_4$ that were prepared by the Signal Corps.

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2. High-power characteristics

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III. J. Green
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